Compression strength of saline water exposed epoxy system containing red mud particles

A THESIS SUBMITTED IN PARTIAL FULFILMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor in Technology

in

Mechanical Engineering

By

SUCHI SMITA NAYAK

Department of Mechanical Engineering

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Under the Guidance of

Prof. S. K. ACHARYA

Department of Mechanical Engineering

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CERTIFICATE

This is to certify that thesis entitled, "COMPRESSION STRENGTH OF SALINE WATER EXPOSED EPOXY SYSTEM CONTAINING RED MUD PARTICLES**"** submitted by Ms. SUCHI SMITA NAYAK in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Mechanical Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma.

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ACKNOWLEDGEMENT

It is with a feeling of great pleasure that I would like to express my most sincere heartfelt gratitude to Prof.S.K.Acharya, Asst. Professor, Dept. of Mechanical Engineering, NIT, Rourkela for suggesting the topic for my thesis report and for his ready and able guidance through out the course of my preparing the report. I am greatly indebted to him for his constructive suggestions and criticism from time to time during the course of progress of my work.

I express my sincere thanks to Prof. R.K.Shaoo, Head of the Department of Mechanical Engineering, NIT, Rourkela for providing me the necessary facilities in the department.

I also express my thanks to Dr. S. C. Mishra of Metallurgical and Materials Engineering Department for helping in the micro structural study in his department.

I express my sincere gratitude to Prof. K.P.Maity, Co-ordinator of M.E. course for his timely help during the course of work.

I thankful to Sri Rajesh Pattnayak and Sri Samir Pradhan for their co-operation in experimental work. I am also thankful to Mr. N.P.Barik and all the staff members of the department of Mechanical Engineering and to all my well wishers for their inspiration and help.

I feel pleased and privileged to fulfill my parents' ambition and I am greatly indepted to them for bearing the inconvenience during my M.E. course. I express my appreciation to my husband for his understanding, patience and active co-operation throughout my M.E. course.

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CONTENTS

ABSRACT

Polymers materials by themselves have found extensive use in non critical products. Such products are used in advanced engineering applications when reinforced with stronger materials. Among the reinforcements, the fibrous variety, whether continuous or discontinuous, occupies a key position. Hence, many inorganic fiber reinforced polymer system have made their entry at various application levels. Because of their large aspect ratio, they yield components, which have anisotropy when the reinforcements are aligned.

In order to secure isentropic properties in composites, reinforcements with near spherical shapes have been tried e.g., glass micro spheres are known as micro balloons. Such man made reinforcements, through yield attractive mechanical properties, are expensive. Hence, a search for cheaper reinforcements is a key subject that needs the attention of material scientists. One such inexpensive filler to fit the slot is red mud. Being a by product of alumina plants, its disposal causes considerable environmental problems. Hence, there is need to tap this inexpensive material for possible use with other system including the polymer based ones. The present study looks at how this pozzolanic red mud, when introduced into a thermoset, responds the exposure to an aqueous medium like laboratory prepared saline water. The results show that when the red mud content in the system is large, the absorption levels are high. The results further revealed that the unexposed samples generally record an increasing strength value with red mud content.

LIST OF TABLES

LIST OF FIGURES

CHAPTER – 1

1.1 BACKGROUND:

Over the last thirty years composite materials, plastics and ceramics have been the dominant emerging materials. The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications.

While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the composites industry. It is obvious, especially for composites, that the improvement in manufacturing technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material, process, tooling, quality assurance, manufacturing, and even program management for composites to become competitive with metals.

The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years.

Unlike conventional materials (e.g., steel), the properties of the composite material can be designed considering the structural aspects. The design of a structural component using composites involves both material and structural design. Composite properties (e.g. stiffness, thermal expansion etc.) can be varied continuously over a broad range of values under the control of the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement.

Whilst the use of composites will be a clear choice in many instances, material selection in others will depend on factors such as working lifetime requirements, number of items to be produced (run length), complexity of product shape, possible savings in assembly costs and on the experience & skills the designer in tapping the optimum potential of composites. In some instances, best results may be achieved through the use of composites in conjunction with traditional materials.

1.2 DEFINITION OF COMPOSITE

The most widely used meaning is the following one, which has been stated by Jartiz [1] “Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form”.

The weakness of this definition resided in the fact that it allows one to classify among the composites any mixture of materials without indicating either its specificity or the laws which should given it which distinguishes it from other very banal, meaningless mixtures.

Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Beghezan [3] defines as “The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short comings”, in order to obtain improved materials.

Van Suchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.3 CLASSIFICATION

Composite materials can be classified in different ways [5]. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in table1.1. The two broad classes of composites are (1) Particulate composites and (2) Fibrous composites.

1.3.1 Particulate Composites

As the name itself indicates, the reinforcement is of particle nature (platelets are also included in this class). It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape, but it is approximately equiaxed. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage.

1.3.2 Fibrous composites

A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might other wise lead to failure, particularly with brittle matrices.

Man-made filaments or fibers of non polymeric materials exhibit much higher strength along their length since large flaws, which may be present in the bulk material, are minimized because of the small cross-sectional dimensions of the fiber. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness.

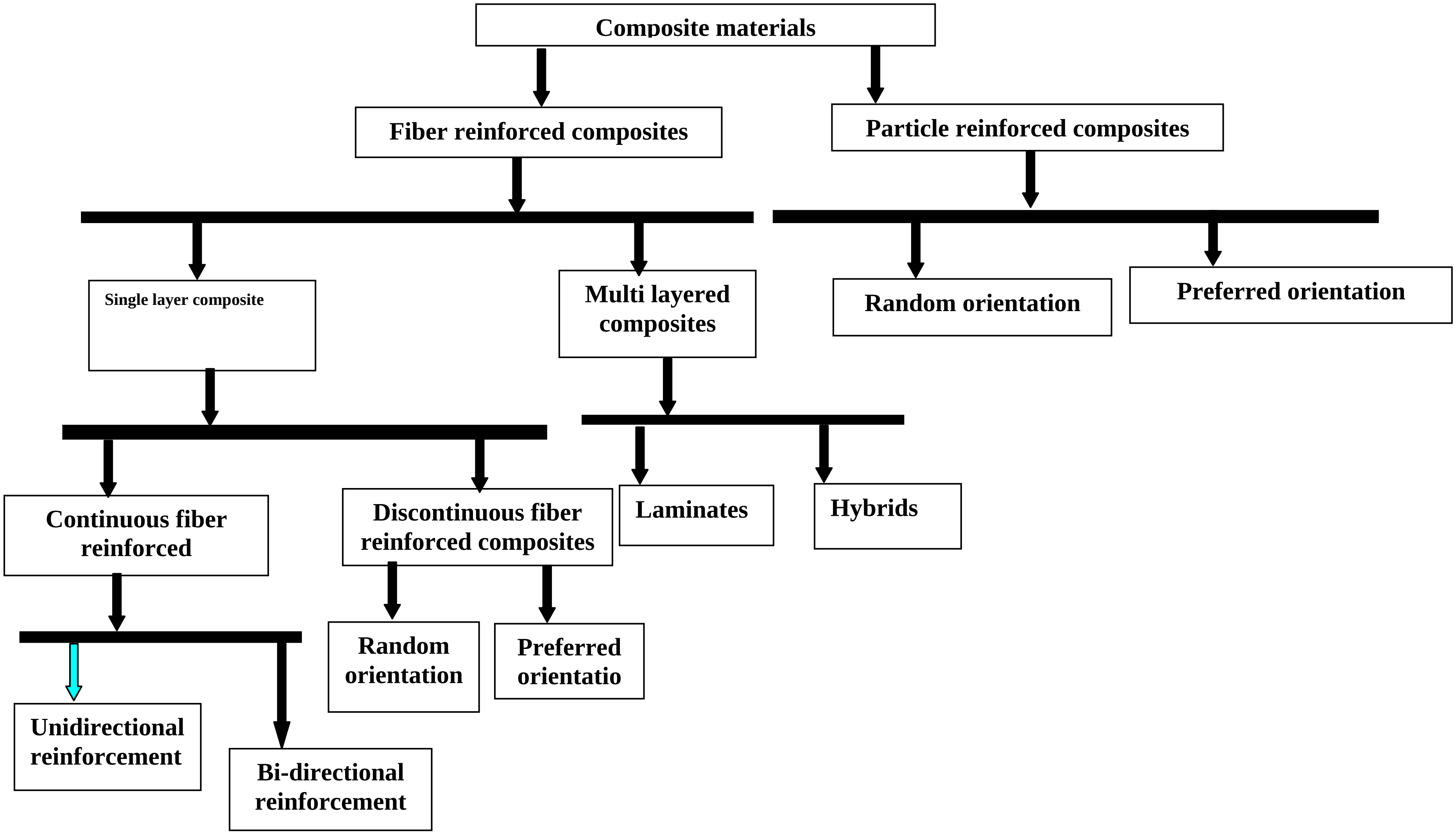

Table: 1 .1 Classification of composites

Fibers, because of their small cross- sectional dimensions, are not directly usable in engineering applications. They are, therefore, embedded in matrix materials to form fibrous composites. The matrix serves to bind the fibers together, transfer loads to the fibers, and protect them against environmental attack and damage due to handling. In discontinuous fiber reinforced composites, the load transfer function of the matrix is more critical than in continuous fiber composites.

1.4 COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the 'matrix'), and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

1.4.1 Role of matrix in a composite

Many materials when they are in a fibrous form exhibit very good strength property but to achieve these properties the fibers should be bonded by a suitable matrix. The matrix isolates the fibers from one another in order to prevent abrasion and formation of new surface flaws and acts as a bridge to hold the fibers in place. A good matrix should possess ability to deform easily under applied load, transfer the load onto the fibers and evenly distributive stress concentration.

1.4.2 Materials used as matrices in composites

(a) BULK PHASES

(1) Metal Matrices

Metal matrix composites possess some attractive properties, when compared with organic matrices. These include (i) strength retention at higher temperatures, (ii) higher transverse strength, (iii) better electrical conductivity, (iv) superior thermal conductivity, (v) higher erosion resistance etc. However, the major disadvantage of metal matrix composites is their higher densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high-energy requirement for fabrication of such composites.

(2) Polymer Matrices

A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites. Some of the major advantages and limitations of resin matrices are shown in Table 1.2.

Table 1.2

Advantages and limitations of polymeric matrix materials

Advantages	Limitations
Low densities	Low transverse strength
Good corrosion resistance	Low operational temperature limits
Low thermal conductivities	
Low electrical conductivities	
Translucence	
Aesthetic Colour effects	

Generally speaking, the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication process selected and also stand up to the service conditions. Apart from these properties, the resin matrix must be capable of wetting and penetrating into the bundles of fibers which provide the reinforcement, replacing the dead air spaces therein and offering those physical characteristics capable of enhancing the performance of fibers.

(3) Ceramic Matrices

Ceramic fibers, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form (e.g. zinc and calcium phosphate). Ceramic Matrix Composites (CMCs) used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibers, or whiskers such as those made from silicon carbide and boron nitride.

(b) REINFORCEMENT

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibers used in composites have different properties and so affect the properties of the composite in different ways. For most of the applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible to achieve different characteristics.

(c) INTERFACE

It has characteristics that are not depicted by any of the component in isolation. The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must “wet” the fiber. Coupling agents are frequently used to improve wettability. Well “wetted” fibers increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the fibers via the interface. This means that the interface must be large and exhibit strong adhesion between fibers and matrix. Failure at the interface (called debonding) may or may not be desirable.

REDMUD-INITIATIVE IN PRODUCT DEVELOPMENT

Production of alumina from Bauxite by the Bayer’s process is associated with generation of red mud as the major waste material. Depending upon the quality of bauxite the quantity of red mud generation varies from 55-65% of the Bauxite processed. This waste material has been accumulating at an increasing rate throughout the world. In India at present, Aluminum production per year is 0.46 million tons of red mud generation in the country. The disposal / utilization of red mud have been an acute problem and a clear– cut solution is not available till date. Different avenues of red mud utilization are more or less known but none of them have so far proved to be economically viable or commercially feasible. However survey of literature on utilization of red mud published so far [1] it is revealed that use of red mud is restricted only for recovery metal values, recovery of alkali, use as catalyst, as building material, in paints& pigments etc. Use of red mud as a reinforcement material for preparation of PMCs has not been reported so far.

In order to find an application to this valuable waste, in this present work an attempt has been made to develop a polymer matrix composite (epoxy resin) using red mud as reinforcement and to study its compressive strength when it is exposed to saline water. The composites are to be prepared with different volume fractions of red mud. The composites are then to be treated with saline water for various time lengths. The change in weight volume and dimensions are to be studied for various treatments. Shear strength of the composites has to be evaluated by three point bend test. Then Micro Structural Examination (SEM) will be made to ascertain the fracture behavior of the composite.

Keeping all this in view the entire work has been divided into four chapters.

In the second chapter work related to present investigations available in literatures are presented.

The third chapter represents the preparation of specimens for the composites, their treatments and characterization.

In fifth conclusions have been drawn from the above studies mentioning the scope for future work.

CHAPTER – 2

2. LITERATURE SURVEY

Polymers materials by themselves have found extensive use in non critical products [1]. Such products are used in advanced engineering applications when reinforced with stronger materials. Among the reinforcements, the fibrous variety, whether continuous or discontinuous, occupies a key position. Hence, many inorganic fiber reinforced polymer system have made their entry at various application levels. Because of their large aspect ratio, they yield components, which have anisotropy when the reinforcements are aligned.

In order to secure isentropic properties in composites, reinforcements with near spherical shapes have been tried [2] e.g., glass micro spheres are known as micro balloons [3, 4]. Such man made reinforcements, through yield attractive mechanical properties, are expensive. Hence, a search for cheaper reinforcements is a key subject that needs the attention of material scientists. One such inexpensive filler to fit the slot is red mud [5]. Being a by product of alumina plants, its disposal causes considerable environmental problems [6]. Hence, there is need to tap this inexpensive material for possible use with other system including the polymer based ones. The present study looks at how this pozzolanic red mud, when introduced into a thermoset, responds the exposure to an aqueous medium like laboratory prepared saline water [7,8].

A perusal of literature shows that little attention has been focused on the compressive property of epoxy system in general and filled ones in particular. Red mud, as it is pozzolanic in nature, forms a cementitious system on absorbing water. Consequently, the mechanical property change. Hence, in the present study, an attempt to monitor one such mechanical property, namely, the compression strength of epoxy resin containing different amount of red mud filler materials has been made. The works also looks at the saline water absorption property of red mud by noting the weight measurements at different intervals of time up to 64hrs. For a structure property correlation, the failure characteristics of such materials on fractured samples were observed under scanning electron microscopy (SEM) following the compression tests.

After reviewing the existing literature available on particulate filler composites, particularly minerals and inorganic oxides (alumina and silica) composites efforts are put to understand the basic needs of the growing composite industry. The conclusions drawn from this is that, the success of combining cheap materials like minerals ores and industrial wastes with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers are cheap and easily available. Moreover, despite their low strength, they can lead to composites with high specific strengths.

Thus the priority of this work is to prepare Polymer Matrix Composites (PMCs) using red mud (waste from alumina industry) as reinforcement material and to study its compression strength behaviour exposed to saline water.

CHAPTER – 3

3. MATERIALS AND METHODS:

3.1 RAW MATERIALS:

Raw materials used in this experimental work are:

1. Epoxy resin
2. Red mud
3. Hardener

3.1.1 Epoxy resin

Softener (Araldite LY 556) made by CIBA GUGYE limited having the following outstanding properties has been used as the matrix material.

a. Excellent adhesion to different materials.
b. High resistance to chemical and atmospheric attack.
c. High dimensional stability.
d. Free from internal stresses.
e. Excellent mechanical and electrical properties.
f. Odorless, tasteless and completely nontoxic.
g. Negligible shrinkage.

3.1.2 Red Mud

The red mud used for the present investigation was brought from the aluminum refinery of NALCO located at Damanjodi, Koraput, Orissa. Dust was prepared manually. The size of the dust was measured by using a sieve. As per this analysis the average size of the dust was 150 micron. The presence of different elements as confirmed by chemical analysis is presented in table - 3.1.

3.1.3 Hardener

In the present work hardener (HY951) is used. This has a viscosity of 10-20 MPa at 25^{o}c

3.2 PREPARATION OF COMPOSITES:

The following procedure has been adopted for the preparation of the specimen

(a) Red mud preparation:-

The required quantities of red mud (10, 20 and 30 percent by weight) were taken in powder containers. The red mud was preheated in a furnace up to 400^{o}C and maintained at that temperature before mixing with epoxy resin.

(b) Composite preparation:-

A wooden mold of dimension (120x100x6) mm was used for casting the composite sheet. The samples were manufactured with 10, 20 and 30 % volume fraction of red mud**.** For different volume fraction of red mud, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. For quick and easy removal of composite sheets, mold release sheet was put over the glass plate and a mold release spray was applied at the inner surface of the mold. After keeping the mold on a glass sheet a thin layer (≈ 2 mm thickness) of the mixture was poured. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. This procedure was adopted for preparation of 10, 20 and 30 % volume fractions of composites. After 72 hrs the samples were taken out of the mold, cut into different sizes and kept in air tight container for further experimentation.

3.3 EXPERIMENTAL PROCEDURE:

3.3.1 Saline Water Exposure

To study the absorption of aqueous medium due to immersion, laboratory-prepared saline water was employed. For this, 4 wt% of NaCl solution was prepared in a beaker. While one batch of samples of each composition was kept as such (i.e., without exposure), the other was immersed in this medium in the beaker maintained at an ambient temperature. The former category formed the unexposed set while the latter the exposed ones, both the sets being subjected to compression tests later on. For monitoring the weight change at periodic intervals of time, the appropriately coded samples, whose dry weights were noted prior to immersing in water, were withdrawn and wiped dry using an absorbent material. They were weighed accurately, at ambient conditions, using an electronic balance in order to note the difference consequent to saline water exposure. Weight readings were taken at regular intervals of time. Every time, five samples were used for weight data gathering and the average value thus determined was used for data analysis. This procedure of aqueous medium data acquisition was repeated for each of the compositions (i.e., 10, 20 and 30%) cast in this work.

3.4 CHARACTERIZATION

3.4.1 Compression Testing

The composites after treated in saline water condition, the compression test was carried out in an UTM 201 machine in accordance with ASTM D2344-84 to measure the flexural strength of the composites. The loading arrangement for the specimen and the photograph of the machine used are shown in fig 3.1. All the specimens (composites) were of rectangular shape having length varied from 100-125 mm, breadth of 100-110 mm and thickness of 4-6 mm. A span of 100 mm was employed maintaining a cross head speed of 10mm/min. The flexural strength and inter laminar shear stress found out from the experiment are presented in table 3.3.

The flexural interlaminar shear strength (ILSS) of the composite which is the maximum shear stress that a material can withstand before it ruptures, was calculated using the equation

$$\sigma m = 3f/4bt$$

Where σm is the ILSS, f is the load, b is the width and t is the thickness of the specimen under test. The maximum tensile stress was found out form the equation.

$$\tau m = 3fl/2bt^2$$

Where τm is the maximum tensile stress and l is the gauge length. A minimum of five samples, as stated earlier, were used for compression tests on any similarly processed samples of different compositions.

Table 3.1 Chemical (dry) analysis of red mud

Constituents	% (wt)	Constituents	% (wt)
Al_2O_3	15.0	Fe_2O_3	54.8
TiO_2	3.7	SiO_2	8.44
Na_2O	4.8	CaO	2.5
P_2O_5	0.67	V_2O_5	0.38
Ga_2O_3	0.096	Mn	1.1
Zn	0.018	Mg	0.056
Organic C	0.88	L.O.I	Balance

Table-3.2

Cumulative Weight Change for neat epoxy, 10%, 20%, and 30% fiber volume fraction composites in SALINE WATER EXPOSURE

Volume fraction of red mud	NE			10%			20%			30%		
Treatment (hrs)	Initial Wt. (gm)	Final Wt. (gm)	Difference (gm)	Initial Wt. (gm)	Final Wt. (gm)	Difference (gm)	Initial Wt. (gm)	Final Wt. (gm)	Difference (gm)	Initial Wt. (gm)	Final Wt. (gm)	Difference (gm)
8	7.92	7.92	0	13.43	13.43	0	16.82	16.82	0	16.82	16.82	0
16	7.92	7.935	0.015	13.43	13.44	0.01	16.82	16.845	0.025	16.82	17.11	0.29
24	7.92	7.936	0.016	13.43	13.456	0.026	16.82	16.864	0.044	16.82	17.19	0.37
32	7.92	7.938	0.018	13.43	13.466	0.036	16.82	16.89	0.07	16.82	17.21	0.39
40	7.92	7.956	0.036	13.43	13.503	0.073	16.82	16.93	0.11	16.82	17.237	0.417
48	7.92	7.975	0.055	13.43	13.541	0.111	16.82	17	0.18	16.82	17.257	0.437
56	7.92	7.996	0.076	13.43	13.607	0.177	16.82	17.07	0.25	16.82	17.27	0.45
64	7.92	8.02	0.1	13.43	13.668	0.238	16.82	17.13	0.31	16.82	17.29	0.47

Table-3.3

Flexural strengths of 10, 20 & 30 % fiber volume fraction composites

Volume fraction of fillers (%)	Flexural Strength (MPa)	
	Dry condition	Weight condition
Neat Epoxy	82	89
10%	83.5	85
20%	92	88
30%	89	74

Fig 3.1 Testing machine with the specimen in loading position

Fig 3.2 Different loading position of the specimen in the Testing Machine

CHAPTER – 4

4.1 RESULT & DISCUSSION:

Fig: 4.1 shows the weight changes the neat epoxy and the composite. It is seen that the weight change in the composite for 30% RM is minimum. There is a large difference between the weight change of neat epoxy and the composite. The rate of swelling for all the composite up to 5hrs follows linearity. The rate of swelling suddenly increases at a very fast rate which can be seen from the plot. The rate of swelling gets affected because of interaction of electron rich species with sodium ions which forms a mono layer at the initial period and prevents swelling. That monolayer probably gets 1 break during subsequent period of exposure and the weight change suddenly increases to a higher value.

Fig:4.2 displays the variation in strength data with respect to red mud content tested in un exposed condition. From the plot it is apparent that a general upward trend for strength is recorded as red mud content in the matrix increases.

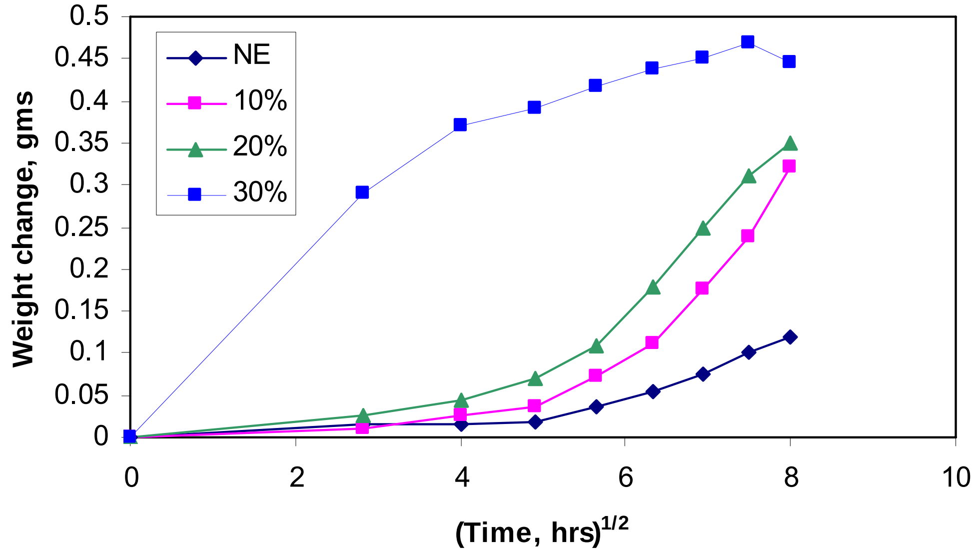

Fig. 4.1 weight changes in neat epoxy 10, 20, 30% filler volume fraction composites subjected to saline water exposure.

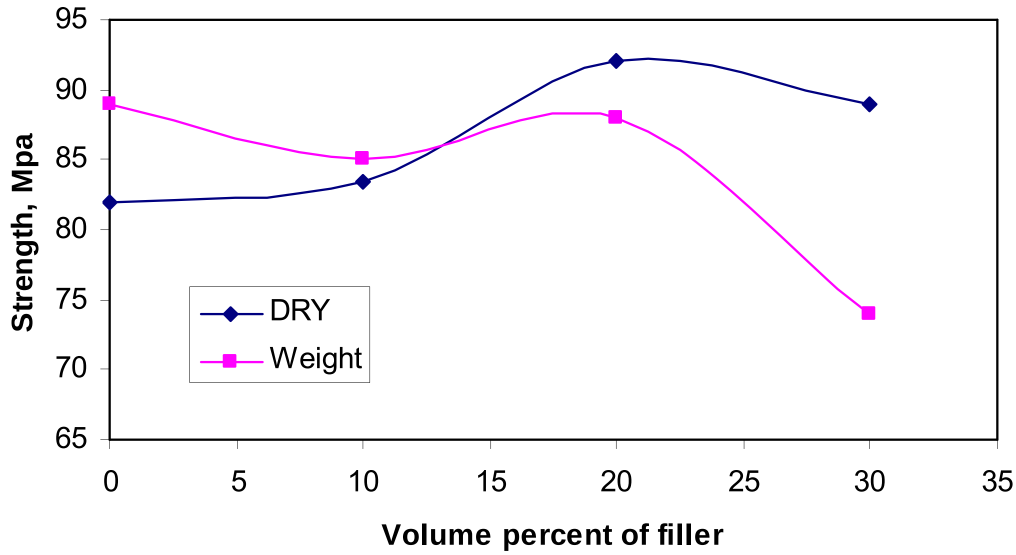

Fig. 4.2 strength of unexposed and exposed samples containing varying amounts of red mud.

4.2. FACTROGRAPHIC ANALYSIS:

To understand how the red mud contribute to strength through a resistance offered to deformation the structural features, as stated earlier and studied using scanning electron microscopy (SEM) on these compression failed samples.
Fig:4.3 shows the unexposed (i.e dry) Neat Epoxy samples. This shows a typical river pattern.

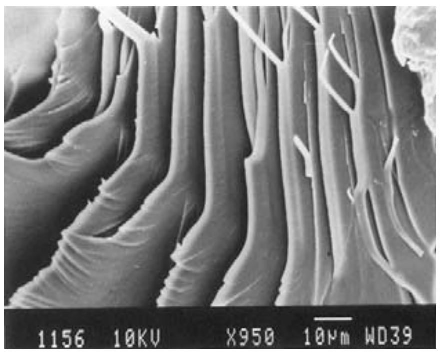

Fig. 4.3 NE sample showing river pattern.

Fig:4.4 shows the 10% RM reinforcements sample is higher than the reinforced sample is higher than the unreinforced one (fig-4.4) which is indicative of resistance offered by the red mud. This is clearly from the deformation bends which are present as curvilinear features marked 1,2,3,4.

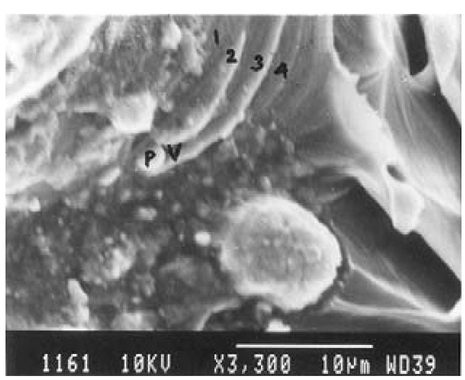

Fig.4.4 10% reinforcement

Fig: 4.5 shows the features recorded for 20% reinforcement. Its strength is higher than RM 10 samples.

The stepped appearance at the center of the micrograph and positioning of red mud particles in the path are responsible for the resistance offered by the system.

As regards the 30% case the closeness of the particles and consequently the ease with which the interface separation leading to failure occur are responsible for a small drop in strength [fig.4.6].

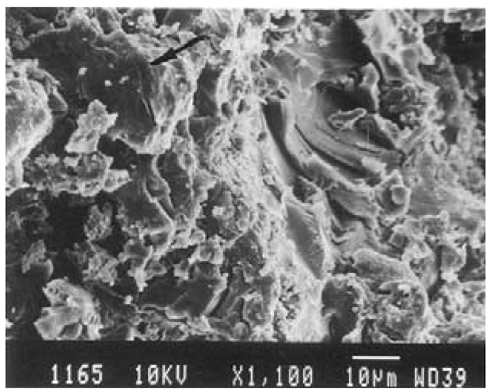

Fig.4.5 20% reinforcement

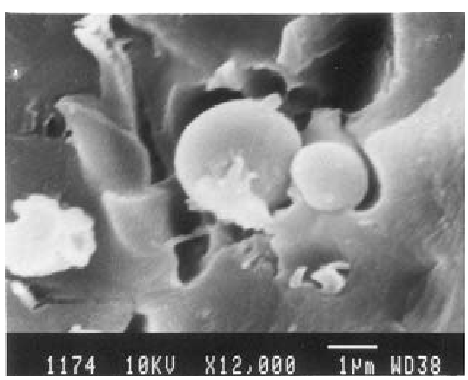

Fig.4.6 30% reinforcement

4.3. **CONCLUSION:**

1) Red mud the bauxite residue can successfully be utilized to manufacture epoxy based composite.
2) The larger the red mud contents in the epoxy the higher the extent of moisture ingression.
3) From the strength consideration it is seen that 20% reinforcement for both dry and saline treated has got optimum strength.
4) From the SEM studies it is clear that interfacial debonding of the red mud particles are responsible for failure in the composite system.

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